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TITLE: Prepn. of thin fullerene films - by forming films on substrates and polymerising using light energy.

PATENT-ASSIGNEE: NEC CORP (NIDE)

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BASIC-ABSTRACT:

Prepn. of thin fullerene films comprises forming thin fullerene films on substrates and irradiating with light energy to polymerise the thin films.

ADVANTAGE - The thin films obtd. consist of fullerene molecules firmly bound together and therefore they do not separate or move on the substrates.

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(71)Applicant : NEC CORP

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(54) PRODUCTION OF FULLERENE THIN FILM

(57)Abstract:

PURPOSE: To form a rigid fullerene thin film by providing a process for forming the fullerene thin film on a substrate and a process for polymerizing the fullerene by the irradiation of light energy to prevent the detachment or movement of the fullerene from the substrate.

CONSTITUTION: The fullerene is made of carbons bonded into a basket state having single bonds and double bonds between carbons. When the fullerene is irradiated with light at the wavelength of about 300nm, a part of the double bonds is broken to bond fullerene molecules to each other. As a result, several numbers of fullereene molecules are cross-linked, and then, fullerene is fixed and so prevented from the detachment from the substrate or the movement on the substrate. In the figure, after a monomolecular layer of C60 is vapor deposited on the (001) plane of a molybdenum disulfide substrate 2 kept at 200°C from a vapor deposition cell 1 under vacuum, a shutter 3 is closed and ultraviolet ray at the wavelength of 300nm is applied through a viewing port 4 for 1min from a ultraviolet lamp 5. The work of vapor depositing the monomolecular layer of C60 and irradiating with light is repeated 100 times.



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CLAIMS

[Claim(s)]

[Claim 1] The fullerene thin-film-fabrication method characterized by having the process which forms a fullerene thin film on a substrate, and the process which gives a light energy to the aforementioned fullerene thin film, and carries out the polymerization of the fullerene in the aforementioned fullerene thin film.

[Claim 2] The fullerene thin-film-fabrication method according to claim 1 characterized by performing the aforementioned fullerene thin film formation process and the aforementioned photopolymerization process by turns.

[Claim 3] The fullerene thin-film-fabrication method according to claim 1 characterized by giving a light energy to the aforementioned fullerene thin film, and carrying out the polymerization of the fullerene in the aforementioned fullerene thin film after forming the fullerene thin film of n molecular layer (n is one or more integers) on a substrate.

[Claim 4] The fullerene thin-film-fabrication method according to claim 1, 2, or 3 characterized by heating a thin film growth substrate in the aforementioned photopolymerization process.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] this invention relates to the thin-film-fabrication method of the fullerene which the carbon atom combined in the shape of a basket.

[0002]

[Description of the Prior Art] When fullerene, such as C₆₀ by which the extensive synthesis method was discovered by Kretschmer etc. in 1990 (Nature, 347,354 (1990)), and C₇₀, introduced alkali metal and alkaline earth metal between C₆₀ molecules under C₆₀ crystal by HADON etc. after that (Nature, 350,320 (1991)), it was discovered that physical properties, such as superconduction, are shown. This has a superconduction transition temperature very high as a molecule superconductor, and the application to a device etc. is expected. In connection with it, importance is attached to thin film-ization of fullerene, and research of thin-film-izing of fullerene is advanced by various thin-film-fabrication methods.

[0003]

[Problem(s) to be Solved by the Invention] Thin-film-fabrication methods, such as an organic molecular-beam vacuum deposition, a vacuum deposition method, and the Langmuire-Brodgett method, were used for the thin-film-fabrication method of old fullerene. However, the thin film made by these methods had problems, like in combination of the matter called Van der Waals force in fullerene, such as C₆₀, since it has joined together by the very weak force, the re-secession from a substrate and fullerene move about on a substrate, and structure is confused. this invention solves such a technical problem and aims at fixing the bonding strength between fullerene so that strength and fullerene may not secede from a substrate or it may not move about on a substrate.

[0004]

[Means for Solving the Problem] The place by which it is characterized [of this invention] is to form the thin film of fullerene, such as C₆₀ and C₇₀, on a substrate, give a light energy to the formed fullerene thin film, cause a chemical reaction between fullerene molecules, and carry out the polymerization of the fullerene molecule comrade by the conventional methods, such as an organic molecular-beam vacuum deposition.

[0005] Invention of the 1st of this invention is the fullerene thin-film-fabrication method characterized by having the process which forms a fullerene thin film on a substrate, and the process which gives a light energy to the aforementioned fullerene thin film, and carries out the polymerization of the fullerene in a fullerene thin film.

[0006] It is the fullerene thin-film-fabrication method given in the 1st invention characterized by the 2nd invention performing the aforementioned fullerene thin film formation process and the aforementioned photopolymerization process by turns.

[0007] The 3rd invention is the fullerene thin-film-fabrication method given in the 1st invention characterized by giving a light energy to the aforementioned fullerene thin film, and carrying out the polymerization of the fullerene in a fullerene thin film, after forming the fullerene thin film of n

molecular layer (n is one or more integers) on a substrate.

[0008] The 4th invention is the fullerene thin-film-fabrication method given in the 1st invention characterized by heating a thin film growth substrate, the 2nd invention, or the 3rd invention in the aforementioned photopolymerization process.

[0009]

[Function] Carbon joins together in the shape of a basket, and fullerene consists of single combination between carbon, and a double bond. If light with a wavelength of about 300nm is irradiated at fullerene, by the light energy, a part of double bond will go out, and fullerene will join together (polymerization). Between a lot of fullerene becomes like meshes of a net by this, and it is fixed, without fullerene's seceding from a substrate or moving about on a substrate.

[0010]

[Example] One example of this invention is shown below with reference to a drawing. Drawing 1 is one example of the equipment used for this invention.

[0011] (Example 1) On the molybdenum disulfide (001) side substrate 2 held at 200 degrees C, under the vacuum of 4×10^{-7} Pa, after depositing C60 of one molecular layer (8A) from the vacuum evaporation cell 1, the shutter 3 was closed, the viewing port 4 was spaced, and ultraviolet rays with a wavelength of 300nm were irradiated for 1 minute from the ultraviolet ray lamp 5. If 1 molecular-layer vacuum evaporation of C60 was carried out, the work which irradiates light was repeated 100 times and C60 thin film of 100 molecular layers (800A) was produced.

[0012] After measuring correctly the thickness of produced C60 polymerization thin film and C60 conventional thin film deposited without performing photopolymerization, Although the thickness of the thin film deposited without performing photopolymerization was 300A when thickness was measured after heating a thin film at 400 degrees C which is the temperature to which C60 evaporates and holding it in a 4×10^{-7} Pa vacuum for 12 hours The thickness of a thin film including a photopolymerization process was 670A. In the photopolymerization thin film by this invention, it was checked that the re-secession from the substrate of C60 molecule by heating is very small.

[0013] moreover, small in C60 thin film which deposited 100 molecular layers of C60 thin films not using the photopolymerization process when continuation observation of the 10micrometerx10micrometer field was carried out with the contact atomic force microscope (it is henceforth described as AFM) -- to an image being confused by several scans, by C60 thin film using the photopolymerization process, even if it performed 50 scans, change was not looked at by the image Although crystallinity will be confused and it will change to an amorphous state as thickness becomes thick if the vacuum evaporation of the fullerene is carried out and it carries out the laminating, without performing photopolymerization like before, since according to this invention the fullerene comrade did the polymerization and is connected firmly, it turns out that the film which does not have almost change in a molecule interval is obtained with the 1st layer also in the thick portion of thickness. In addition, the firmer film was obtained, when performing photopolymerization and substrate temperature was heated at about 150 degrees C.

[0014] (Example 2) On the quartz-glass substrate 2 held at 100 degrees C, after depositing C60 of one molecular layer (8A) from the vacuum evaporation cell 1 under the vacuum of 4×10^{-7} Pa, the shutter 3 was closed, the viewing port 4 was spaced, and ultraviolet rays with a wavelength of 300nm were irradiated for 1 minute from the ultraviolet ray lamp 5. If 1 molecular-layer vacuum evaporation of C60 was carried out, the work which irradiates light was repeated 100 times, and C60 thin film of 100 molecular layers (800A) was produced. After measuring correctly the thickness of C60 thin film deposited without performing C60 polymerization thin film and photopolymerization which were produced, Although the thickness of the thin film deposited without performing photopolymerization was 430A when thickness was measured after heating a thin film at 400 degrees C which is the temperature to which C60 evaporates and holding it in a 4×10^{-7} Pa vacuum for 12 hours The thickness of a thin film including a photopolymerization process is 720A, and it was checked in the photopolymerization thin film that the re-secession from the substrate of C60 molecule by heating is very small. Moreover, at C60 thin film which deposited 100 molecular layers of C60 thin films not using

the photopolymerization process when continuation observation of the 10micrometerx10micrometer field was carried out by contacted type AFM, it was small, or to an image being confused by several scans, by C60 thin film using the photopolymerization process, even if it performed 50 scans, change was not looked at by the image. These things showed that C60 comrades in C60 thin film were connected firmly. In addition, the firmer film was obtained, when performing photopolymerization and substrate temperature was heated at about 150 degrees C.

[0015] (Example 3) On the graphite (001) side substrate 2 held at 200 degrees C, after depositing C60 of one molecular layer (8A) from the vacuum evaporatio cell 1 under the vacuum of 4×10^{-7} Pa, the shutter 3 was closed, the viewing port 4 was spaced, and ultraviolet rays with a wavelength of 300nm were irradiated for 1 minute from the ultraviolet ray lamp 5. If 1 molecular-layer vacuum evaporatio of C60 was carried out, the work which irradiates light was repeated 100 times and C60 thin film of 100 molecular layers (800A) was produced. After measuring correctly the thickness of C60 thin film deposited without performing C60 polymerization thin film and photopolymerization which were produced, Although the thickness of the thin film deposited without performing photopolymerization was 300A when thickness was measured after heating a thin film at 400 degrees C which is the temperature to which C60 evaporates and holding it in a 4×10^{-7} Pa vacuum for 12 hours The thickness of a thin film including a photopolymerization process is 670A, and it was checked that a photopolymerization thin film has the very small re-secession from the substrate of C60 molecule by heating. Moreover, at C60 thin film which deposited 100 molecular layers of C60 thin films not using the photopolymerization process when continuation observation of the 10micrometerx10micrometer field was carried out by contacted type AFM, it was small, or to an image being confused by several scans, by C60 thin film using the photopolymerization process, even if it performed 50 scans, change was not looked at by the image. These things showed that C60 comrades in C60 thin film were connected firmly. In addition, the firmer film was obtained, when performing photopolymerization and substrate temperature was heated at about 150 degrees C.

[0016] (Example 4) On the molybdenum disulfide (001) side substrate 2 held at 200 degrees C, after depositing C70 of one molecular layer (9A) from the vacuum evaporatio cell 1 under the vacuum of 4×10^{-7} Pa, the shutter 3 was closed, the viewing port 4 was spaced, and ultraviolet rays with a wavelength of 300nm were irradiated for 1 minute from the ultraviolet ray lamp 5. If 1 molecular-layer vacuum evaporatio of C70 was carried out, the work which irradiates light was repeated 100 times and C70 thin film of 100 molecular layers (900A) was produced. After measuring correctly the thickness of C70 thin film deposited without performing C70 polymerization thin film and photopolymerization which were produced, Although the thickness of the thin film deposited without performing photopolymerization was 300A when thickness was measured after heating a thin film at 450 degrees C which is the temperature to which C70 evaporates and holding it in a 4×10^{-7} Pa vacuum for 12 hours The thickness of a thin film including a photopolymerization process is 670A, and it was checked that a photopolymerization thin film has the very small re-secession from the substrate of C70 molecule by heating. Moreover, when continuation observation of the 10micrometerx10micrometer field was carried out for C70 thin film by contacted type AFM, it was small, or even if it performed 50 scans, by C70 thin film using the photopolymerization process, change was not looked at by the image to an image being confused by several scans at C70 thin film deposited 100 molecular layers, without using a photopolymerization process. These things showed that C60 comrades in C70 thin film were connected firmly. In addition, the firmer film was obtained, when performing photopolymerization and substrate temperature was heated at about 150 degrees C.

[0017] In the above example, although the thin film formation process and the photopolymerization process were performed by turns as light was irradiated, if 1 molecular-layer vacuum evaporatio of the fullerene was carried out, after carrying out n molecular-layer (n is one or more integers) deposition of the fullerene, you may add and carry out the polymerization of the light energy.

[0018] Also in this case, like examples 1-4, the re-secession from the substrate of the fullerene by heating was very small, and change was not looked at by the image by AFM observation, either.

[0019]

[Effect of the Invention] Like, if the method of this invention is used, a firm fullerene thin film without re-secession of the fullerene from a substrate, fullerene moving about on a substrate and structure being confused, etc. above can be obtained. When applying industrially, it is very important to make a quality and firm thin film.

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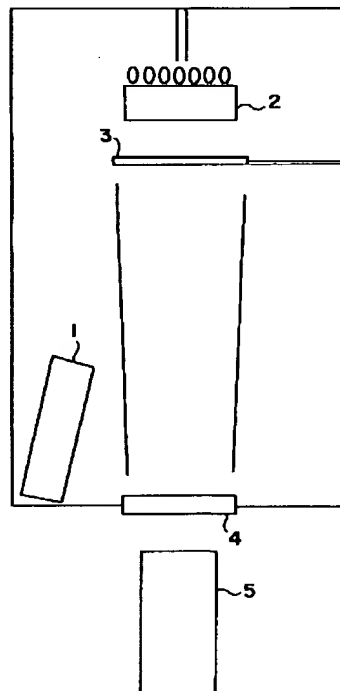
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(54)【発明の名称】 フラーレン薄膜製造方法

(57)【要約】

【目的】 フラーレン間の結合力を強め、フラーレンが基板上で動き回らないように固定されたフラーレン薄膜の製造方法。

【構成】 フラーレン系薄膜の作製において、1分子層のフラーレンを蒸着すると共に光エネルギーを与えフラーレン分子同士を重合させ、フラーレン分子間を網の目のようにし、これを繰り返して強固なフラーレン薄膜を得る。



【特許請求の範囲】

【請求項1】 基板上にフラーレン薄膜を形成する工程と、前記フラーレン薄膜に光エネルギーを与え、前記フラーレン薄膜中のフラーレンを重合する工程を有することを特徴とするフラーレン薄膜製造方法。

【請求項2】 前記フラーレン薄膜形成工程と前記光重合工程を交互に行うことを特徴とする請求項1記載のフラーレン薄膜製造方法。

【請求項3】 基板上にn分子層（nは1以上の整数）のフラーレン薄膜を形成した後に、前記フラーレン薄膜に光エネルギーを与え、前記フラーレン薄膜中のフラーレンを重合することを特徴とする請求項1記載のフラーレン薄膜製造方法。

【請求項4】 前記光重合工程において、薄膜成長基板を加熱することを特徴とする請求項1または請求項2または請求項3記載のフラーレン薄膜製造方法。

【発明の詳細な説明】

【0001】

【産業上の利用分野】本発明は、炭素原子が籠状に結合したフラーレンの薄膜製造方法に関する。

【0002】

【従来の技術】1990年にクレッチマー等（Nature, 347, 354（1990））によって大量合成法が発見されたC₆₀やC₇₀などのフラーレンは、その後、ハドン等（Nature, 350, 320（1991））によってC₆₀結晶中のC₆₀分子間にアルカリ金属やアルカリ土類金属を導入する事により超伝導などの物性を示す事が発見された。これは分子超伝導体としては非常に高い超伝導転移温度を有し、デバイスなどへの応用が期待される。それに伴いフラーレンの薄膜化が重要視され、各種薄膜製造法により、フラーレンの薄膜化の研究が進められている。

【0003】

【発明が解決しようとする課題】これまでのフラーレンの薄膜製造法には有機分子線蒸着法、真空蒸着法、Langmuir-Brodgett法などの薄膜製造法が用いられていた。しかし、これらの方法によって作られた薄膜は、C₆₀などのフラーレンがファンデアワールス力という、物質の結合では非常に弱い力で結合しているために、基板からの再離脱やフラーレンが基板上で動き回り構造が乱れるなどの問題があった。本発明は、このような課題を解決し、フラーレン間の結合力を強め、フラーレン類が基板から離脱したり基板上で動き回ることをないように固定する事を目的としたものである。

【0004】

【課題を解決するための手段】本発明の特徴とするところは、有機分子線蒸着法など従来の方法により、基板上にC₆₀やC₇₀などのフラーレンの薄膜を形成し、形成されたフラーレン薄膜に光エネルギーを与え、フラーレン分子間に化学反応を起こし、フラーレン分子同士を重合

させることにある。

【0005】本発明の第1の発明は、基板上にフラーレン薄膜を形成する工程と、前記フラーレン薄膜に光エネルギーを与え、フラーレン薄膜中のフラーレンを重合する工程を有することを特徴とするフラーレン薄膜製造方法である。

【0006】第2の発明は、前記フラーレン薄膜形成工程と前記光重合工程を交互に行うことを特徴とする第1の発明に記載のフラーレン薄膜製造方法である。

【0007】第3の発明は、基板上にn分子層（nは1以上の整数）のフラーレン薄膜を形成した後に、前記フラーレン薄膜に光エネルギーを与え、フラーレン薄膜中のフラーレンを重合することを特徴とする第1の発明に記載のフラーレン薄膜製造方法である。

【0008】第4の発明は、前記光重合工程において、薄膜成長基板を加熱することを特徴とする第1の発明または第2の発明または第3の発明に記載のフラーレン薄膜製造方法である。

【0009】

【作用】フラーレンは、炭素が籠状に結合し、炭素間の一重結合と二重結合からなる。フラーレンに300nm程度の波長の光を照射すると光エネルギーによって二重結合が一部切れ、フラーレン同士が結合（重合）する。これにより、いくつものフラーレン間が網の目のようになり、フラーレンが基板から離脱したり基板上で動き回ることなく固定される。

【0010】

【実施例】本発明の一実施例を図面を参照して以下に示す。図1は本発明に使用する装置の一実施例である。

【0011】（実施例1）200℃に保持した二硫化モリブデン（001）面基板2上に、 4×10^{-7} Paの真空中で、蒸着セル1から1分子層（8オングストローム）のC₆₀を蒸着した後、シャッター3を閉じ、ビューイングポート4を透して紫外線ランプ5から300nmの波長の紫外線を1分間照射した。C₆₀を1分子層蒸着しては光を照射する作業を100回繰り返して100分子層（800オングストローム）のC₆₀薄膜を作製した。

【0012】作製されたC₆₀重合薄膜と、光重合を行わずに蒸着した従来のC₆₀薄膜の膜厚を正確に測定した後、薄膜を 4×10^{-7} Pa真空中でC₆₀が蒸発する温度である400℃に加熱し、12時間保持した後、膜厚を測定したところ、光重合を行わずに蒸着した薄膜の膜厚は300オングストロームであったが、光重合工程を含む薄膜の膜厚は670オングストロームであった。本発明による光重合薄膜では、加熱によるC₆₀分子の基板からの再離脱が非常に小さい事が確認された。

【0013】また、C₆₀薄膜を接触原子間力顕微鏡（以後AFMと記述する）で10μm×10μmの領域を連続観察したところ、光重合プロセスを用いず100分子

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層蒸着したC₆₀薄膜では、僅か数回の走査で像が乱れてくるのに対し、光重合プロセスを用いたC₆₀薄膜では50回の走査を行っても像に変化が見られなかった。従来のように光重合を行わずにフラーレンを蒸着し積層していくと、膜厚が厚くなるにつれ、結晶性が乱れアモルファス状態へと変化してゆくが、本発明によれば、フラーレン同志が重合し、強固に結び付いているため、膜厚の厚い部分でも、1層目と分子間隔にほとんど変化の無い膜が得られることがわかる。なお、光重合を行う際に基板温度を150℃程度で加熱しておくことにより強固な膜が得られた。

【0014】(実施例2) 100℃に保持した石英ガラス基板2上に、 4×10^{-7} Paの真空下で蒸着セル1から1分子層(8オングストローム)のC₆₀を蒸着した後、シャッター3を閉じ、ビューイングポート4を透して紫外線ランプ5から300 nmの波長の紫外線を1分間照射した。C₆₀を1分子層蒸着しては光を照射する作業を100回繰り返して、100分子層(800オングストローム)のC₆₀薄膜を作製した。作製されたC₆₀重合薄膜と光重合を行わずに蒸着したC₆₀薄膜の膜厚を正確に測定した後、薄膜を 4×10^{-7} Pa真空中でC₆₀が蒸発する温度である400℃に加熱し、12時間保持した後、膜厚を測定したところ、光重合を行わずに蒸着した薄膜の膜厚は430オングストロームであったが、光重合工程を含む薄膜の膜厚は720オングストロームであり、光重合薄膜では加熱によるC₆₀分子の基板からの再離脱が非常に小さい事が確認された。また、C₆₀薄膜を接触型AFMで10 μm×10 μmの領域を連続観察したところ、光重合プロセスを用いず100分子層蒸着したC₆₀薄膜では、僅か数回の走査で像が乱れてくるのに対し、光重合プロセスを用いたC₆₀薄膜では50回の走査を行っても像に変化が見られなかった。これらの事からC₆₀薄膜中のC₆₀同士が強固に結び付いている事が分かった。なお、光重合を行う際に基板温度を150℃程度で加熱しておくことにより強固な膜が得られた。

【0015】(実施例3) 200℃に保持したグラファイト(001)面基板2上に、 4×10^{-7} Paの真空下で蒸着セル1から1分子層(8オングストローム)のC₆₀を蒸着した後、シャッター3を閉じ、ビューイングポート4を透して紫外線ランプ5から300 nmの波長の紫外線を1分間照射した。C₆₀を1分子層蒸着しては光を照射する作業を100回繰り返して100分子層(800オングストローム)のC₆₀薄膜を作製した。作製されたC₆₀重合薄膜と光重合を行わずに蒸着したC₆₀薄膜の膜厚を正確に測定した後、薄膜を 4×10^{-7} Pa真空中でC₆₀が蒸発する温度である400℃に加熱し、12時間保持した後、膜厚を測定したところ、光重合を行わずに蒸着した薄膜の膜厚は300オングストロームであったが、光重合工程を含む薄膜の膜厚は670オングストロームであり、光重合薄膜は加熱によるC₆₀分子の基

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板からの再離脱が非常に小さい事が確認された。また、C₆₀薄膜を接触型AFMで10 μm×10 μmの領域を連続観察したところ、光重合プロセスを用いず100分子層蒸着したC₆₀薄膜では、僅か数回の走査で像が乱れてくるのに対し、光重合プロセスを用いたC₆₀薄膜では50回の走査を行っても像に変化が見られなかった。これらの事からC₆₀薄膜中のC₆₀同士が強固に結び付いている事が分かった。なお、光重合を行う際に基板温度を150℃程度で加熱しておくことにより強固な膜が得られた。

【0016】(実施例4) 200℃に保持した二硫化モリブデン(001)面基板2上に、 4×10^{-7} Paの真空下で蒸着セル1から1分子層(9オングストローム)のC₇₀を蒸着した後、シャッター3を閉じ、ビューイングポート4を透して紫外線ランプ5から300 nmの波長の紫外線を1分間照射した。C₇₀を1分子層蒸着しては光を照射する作業を100回繰り返して100分子層(900オングストローム)のC₇₀薄膜を作製した。作製されたC₇₀重合薄膜と光重合を行わずに蒸着したC₇₀薄膜の膜厚を正確に測定した後、薄膜を 4×10^{-7} Pa真空中でC₇₀が蒸発する温度である450℃に加熱し、12時間保持した後、膜厚を測定したところ光重合を行わずに蒸着した薄膜の膜厚は300オングストロームであったが、光重合工程を含む薄膜の膜厚は670オングストロームであり、光重合薄膜は加熱によるC₇₀分子の基板からの再離脱が非常に小さい事が確認された。また、C₇₀薄膜を接触型AFMで10 μm×10 μmの領域を連続観察したところ、光重合プロセスを用いず100分子層蒸着したC₇₀薄膜では、僅か数回の走査で像が乱れてくるのに対し、光重合プロセスを用いたC₇₀薄膜では50回の走査を行っても像に変化が見られなかった。これらの事からC₇₀薄膜中のC₆₀同士が強固に結び付いている事が分かった。なお、光重合を行う際に基板温度を150℃程度で加熱しておくことにより強固な膜が得られた。

【0017】以上の実施例では、フラーレンを1分子層蒸着しては光を照射するというように、薄膜形成工程と光重合工程とを交互に行ったが、フラーレンをn分子層(nは1以上の整数)蒸着した後に、光エネルギーを加え重合させてもよい。

【0018】この場合にも、実施例1～4と同様に、加熱によるフラーレンの基板からの再離脱が非常に小さく、また、AFM観察によっても像に変化は見られなかった。

【0019】

【発明の効果】以上述べたように、本発明の方法を用いれば、基板からのフラーレンの再離脱や、フラーレンが基板上で動き回り構造が乱れることなどのない強固なフラーレン薄膜を得る事が出来る。工業的に応用する場合、高品質で強固な薄膜を作る事は非常に重要である。

【図面の簡単な説明】

【図1】本発明に用いる装置の一実施例で、装置の構成を示す図である。

【符号の説明】

1 蒸着セル

2 蒸着基板

3 シャッター

4 ビューイングポート

5 紫外線ランプ

【図1】

